Modeling of Beryllia Ceramics Formation Process

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Abstract. Experimental and simulation results of the motion and heat exchange of the beryllia slurry in the annular cavity are presented. The slurry is a highly concentrated structured system where the mineral phase is beryllia powder and the liquid phase is an organic binder (paraffin, oleic acid and beeswax). Mathematical model describes motion and heat exchange of the liquid thermoplastic slurry of beryllia including the aggregate state change. The results of experiments and calculations show the process of molding of the slurry in the annular cavity. The obtained temperature field determines the transition of the slurry from liquid (viscous–plastic) to solid–plastic state. Calculated data of the isotherms of solidication zone of the thermoplastic slurry are in agreement with the experimental results.

Keywords: Thermoplastic slurry, Molding, Solidication Process, Heat Transfer

1 Introduction

The technology of hot pressure molding [1, 2] remains the basis for obtaining long-length, multi-channel, and complex shaped ceramics from non-plastic powders, in spite of using isostatic pressing. Nowadays technology of slurry casting (extrusion) is very relevant in connection with intensive development of MIM technology [3–8], where similar physical processes take place.

While a lot of attention has been paid to improve the technology and to create new equipment in previous years, obtaining fault free products by this method remains an unsolved problem up to now. As a result, the desired quality of moldings and obtaining of acceptable products often can not be achieved, which makes in practice this process low protable. Obtaining ceramic fabrications by hot molding from dispersion materials with anomalous physical properties, such as beryllia (BeO), is particularly complicated. In this case, the difficulties of obtaining the high quality products are caused firstly by thermal properties of BeO, in particular, its unique thermal conductivity. Beryllia ceramic exhibits the highest thermal conductivity among all ceramic materials used in contemporary electronics, new fields of technology and special instrument building [9, 10].

Clearly, it is impossible to eliminate technological limitations and problems without the development based on all experience and knowledge of theoretical representations about regularities and mechanisms of regulation of the thermal conditions on the forming process of molding.

The results of the experiment and the generalizations by calculations of mathematical model of molding process of the beryllia thermoplastic slurry are presented in this paper.

2 Experimental research of the solidication process of molding

Experimental research of the effect of casting conditions on the temperature field in the zone of solidication of the molding was made on the experimental bushing (Fig. 1), by measuring the temperature using a thermocouple installed on the different levels by the height of crystallizer. Experimental bushing is designed for casting of circular tube with the outer diameter 0.02 m and the inner diameter 0.012 m. Material of mandrel and crystallizer is steel of grade X18H10T. The total height of the cylindrical part of the annular cavity is H = 0.028 m, the height of the hot zone of the annular cavity is $h_1 = 0.008$ m, the height of the cold zone of the annular cavity is $h_2 = 0.020$ m. Water with the temperature $t_1 = 80°$C is fed to the upper contour of the crystallizer. Water with the temperature $t_2 =$ 15-20°C is fed to the bottom cold contour. The maximum water flow capacity of the crystallizer is 1500 l/hour.

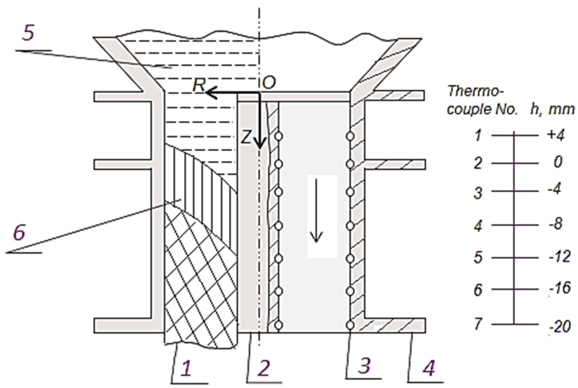

Fig. 1. Scheme of molding solidification in the bushing. (*1 – molding, 2 – mandrel, 3 – thermocouple, 4 – bushing, 5 – liquid slurry, 6 – zone of solidification*).

Conical input part of the bushing is connected with the working tank of casting installation where BeO slurry is kept. Slurry flows to the conical inlet of the annular cavity with an initial temperature of $t_0 = 80°C$. Slurry changes aggregate state and solidifies in result of heat exchange with the walls of mandrel and bushing.

According to the experimental data, dependence of position of the boundary of solidification slurry on different parameters of casting was constructed. The shape of the curve of surface solidification, which is dependent on the casting parameter, is defined by taking into account that the temperature changes linearly by the height and radius of crystallizer on the short segments.

The inuence of molding speed on the thermal regime of the casting control was determined in the first series of experiments. The flow rate and temperature values of hot and cold water in the cooling contours are presented in Table 1. Fig. 2 shows positions of solidification zones depending on the molding speed. Isotherm of the AB corresponds to the temperature 54°C and isotherm of the CD to 40°C (Fig. 2).

Table 1. Regimes of experiments as a function of the molding speed

Number of the diagram		1	2	3	4	5
Molding regime	Hot water flow rate, l/hour	500	500	500	500	500
	Cold water flow rate, l/hour	1500	1500	1500	1500	1500
	Molding speed, mm/min	20	40	60	80	100
	Hot water temperature, °C	80	80	80	80	80
	Cold water temperature, °C	20	20	20	20	20

The increase of the molding speed leads to expansion of solidication zone and its movement to the area of heat extraction of cold contour (Fig. 2). It explains that with increase of molding speed, heat extraction on the walls of the annular cavity does not have time to cool the slurry, and solidification zone extends, and it moves downstream.

In the second series of experiments the influence of cold water temperature and flow rate on the thermal regime of molding solidification was investigated (Table 2).

Table 2. Regimes of experiments as a function of the cold water flow rate and temperature

Number of the diagram		1	2	3	4	5
Molding regime	Hot water flow rate, l/hour	500	500	500	500	500
	Cold water flow rate, l/hour	1000	500	2500	1500	250
	Molding speed, mm/min	20	20	20	20	20
	Hot water temperature, °C	80	80	80	80	80
	Cold water temperature, °C	15	15	15	20	20

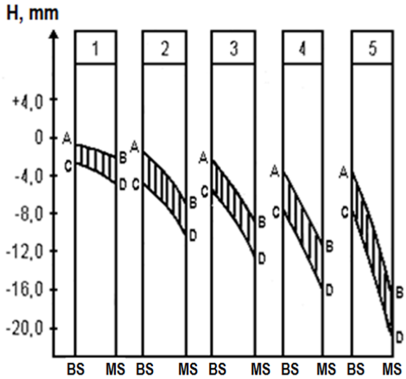

Fig. 2. The position of solidification zone depending on the molding speed. (*AB – the solidus line (54 °C), CD – the solidus line (40 °C), BS – the surface of the bushing, MS – the surface of the mandrel*).

The results of the second series of experiments are shown in Fig. 3. Solidification zones of molding are located in the area of cold contour, and move downstream with reducing cold water flow rate. Reducing of cold water flow rate leads to reduction of the heat extraction on the wall of bushing (Fig. 3).

In the fourth experiment, the beginning of solidification zone is located nearer to the hot contour area. Increase of cold water temperature leads to reduction of temperature difference of the hot slurry and cold water. In the fifth experiment parallel with the increase of cold water temperature reduction of its flow rate takes place (Table 2). Therefore, heat extraction reduces and solidification zone of molding shifts down (Fig. 3).

Accordingly, the experimental data show that the thermal regime of molding of the beryllia thermoplastic slurry is sensitive to change of the molding speed and to heat extraction conditions on the walls of the annular cavity.

3 Mathematical model of the solidification process

Motion and heat exchange of the beryllia thermoplastic slurry in the annular cavity are considered. The slurry flows into cavity with initial temperature of t_0=80°C (Fig. 1). As it moves the slurry is cooled and solidified, and on the output from the cavity it acquires structural form of the tube. The slurry flows in the laminar regime. Due to high viscosity of thermoplastic slurry, Prandtl number is much higher than one. Density of the slurry mass is a function of temperature and it increases while solidification process.

The problem is considered in Cartesian coordinate system with axis z and r. OZ axis coincides with the cavity axis direction, and OR axis is radially directed to it (r_1, radius of the mandrel; r_2, radius of the bushing; r_2–r_1, thickness of the annular cavity). Molding speed is directed vertically downward along the

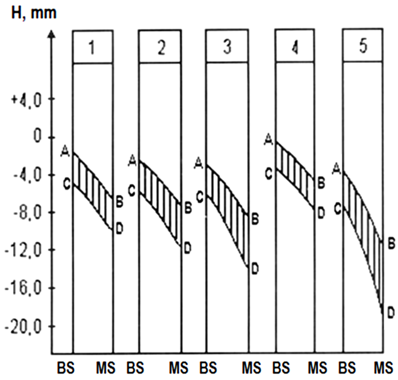

Fig. 3. The position of solidification zone depending on the flow rate and the temperature of cold water. (*AB – the solidus line (54 °C), CD – the solidus line (40 °C), BS – the surface of the bushing, MS – the surface of the mandrel*).

OZ axis. Radial component of the slurry velocity originates due to the heat exchange of the slurry with the walls of annular cavity.

Rheological properties of the slurry change with temperature. The heat of phase transition is released during the change of state. Cooling of the slurry may lead to the irregularity of the temperature profile and rheological properties of the pressing molding. Solidification begins at the walls of the annular cavity, while in the central part slurry may be in liquid state. As a result, in-feeding of slurry for the compensation of internal shrinkage of volume in the cooling zone of the cavity may occur.

According to the experimental data, the slurry solidification occurs at the temperature range from 54 to 40°C. Binder of the slurry is in the amorphous state and passes from the liquid amorphous state to the solid–plastic amorphous state in the zone of solidification [11–14]. The total amount of heat released per unit mass of the slurry mass is determined by the change of enthalpy H at the phase transition zone.

Heat capacity of the slurry changes in the transition zone. Increase of the enthalpy during the phase transition can be determined by the apparent heat capacity method [15–19]. In this method, the latent heat is taken into account by increasing the heat capacity in the phase transition zone. Changing of heat capacity can be represented as [15, 16]:

$$c_p = \begin{cases} c_s, & t < t_s \quad \textit{solid phase} \\ c_{in}, & t_s \le t \le t_l \quad \textit{transition zone} \\ c_l, & t > t_l \quad \textit{liqiud phase} \end{cases} \tag{1}$$

where $A_{in} = \frac{\left\{\int_{t_s}^{t_l} c(t)dt + H_{1\to2}\right\}}{(t_l - t_s)}$, $H_{1\to2}$ – the phase transition specific enthalpy of beryllia slurry with binder mass fraction of $\omega = 0.117$ is determined by experimental data and is equal to $H_{1\to2}$=7800 J/kg [20].

In [17, 18] it is believed that the temperature in the transition zone changes linearly, the expression of the specific heat is defined as:

$$c_p = c(t) + H_{1\to 2}\frac{\partial f_{sl}}{\partial t} \tag{2}$$

$$f_{sl} = \begin{cases} 0, \quad t < t_s \qquad \textit{solid phase} \\ \frac{t-t_s}{t_l-t_s}, \quad t_s \le t \le t_l \quad \textit{transition zone} \\ 1, \quad t > t_l \quad \textit{liquid phase} \end{cases}$$

In [19] phase transition function $\alpha(\bar{t})$ is introduced to the transition zone to consider the latent heat, and changing of the slurry heat capacity is expressed by:

$$c_p = c_s \cdot (1 - \alpha(\bar{t})) + c_l \cdot \alpha(\bar{t}) + H_{1\to 2}\frac{d\alpha}{dt} \tag{3}$$

where c_s – specific heat of the slurry in the solid state, c_l – specific heat of the slurry in the liquid state, $\alpha(\bar{t})= 0$ for the pure solid slurry and $\alpha(\bar{t})= 1$ for the pure liquid slurry, $\bar{t}$ – dimensionless temperature of slurry.

According to the experimental data of beryllia slurry with binder mass fraction of $\omega = 0.117$ function $\alpha(\bar{t})$ takes a form $\alpha(\bar{t}) = 5.712 \cdot \bar{t} - 2.8544$.

The equations (1) – (3) of the method of apparent heat capacity include the latent heat of the phase transition, and are convenient for calculations. For convenience position of the transition zone is not known in advance and is determined as a result of the calculations.

The rheological properties of the slurry for $\omega = 0.117$ depend on temperature [21]:

$$\mu\left(t\right) = 293.6259 \cdot \exp\left(-0.05816 \cdot t\right), \ \ (Pa \cdot s) \tag{4}$$

$$\tau_0\left(t\right) = 11.4 + 11.41 \cdot \exp\left(-\frac{(t-70.05)}{5.47}\right), \ \ (Pa) \tag{5}$$

Density of the thermoplastic slurry is defined by the concentrations of the beryllium oxide powder and the binder:

$$\rho = \frac{\rho_{BeO} \cdot \rho_{bin}}{((1-\omega)\,\rho_{bin} + \omega \cdot \rho_{BeO})}, \quad (g/cm^3) \tag{6}$$

where ρ_{BeO} is the density of the BeO powder, ρ_{bin} is the density of the binder, ω is relative mass content of the binder in the fractions.

The density of the binder where $\omega = 0.117$ is determined by Eq. (7):

$$\rho_{bin}\left(t\right) = 0.852 + 0.0725 \cdot cos\left(0.0561 \cdot (t + 273.15) - 16.7361\right), \quad (g/cm^3) \tag{7}$$

The density of the beryllium oxide is $\rho_{BeO} = 3.02\ g/cm^3$. The density of the binder ρ_{bin} in the range of temperature from $t = 80 - 40$ ℃ changed within

0.7797 to 0.9010 g/cm^3 and the density of the thermoplastic slurry during solidification increases from 2.2457 to 2.3553 g/cm^3 for the fraction $\omega = 0.117$.

Thermal conductivity of the slurry the binder mass fraction $\omega = 0.117$ has the following form [20]:

$$\lambda = 1.6 + 4.8 \cdot \exp\left(-0.017 \cdot t\right), \quad W/(m \cdot ℃) \tag{8}$$

In the experiments [11, 14] beryllia slurry shows thixotropic properties of non-Newtonian fluid, and is described by Shvedov-Bingham rheological model [22]. The motion of the slurry in annulus is considered to be steady-state and the system of equations in the narrow channel is used for its study [23, 24]:

$$\rho u \frac{\partial u}{\partial z} + \rho v \frac{\partial u}{\partial r} = -\frac{dp}{dz} + \frac{1}{r}\frac{\partial}{\partial r}\left(r\mu \frac{\partial u}{\partial r}\right) - \frac{1}{r}\frac{\partial}{\partial r}(r\tau_0) \tag{9}$$

$$\frac{\partial \rho u}{\partial z} + \frac{1}{r}\frac{\partial r \rho v}{\partial r} = 0 \tag{10}$$

In the limit of solid-plastic state of the slurry the motion equation (9) expresses the squeezing-out of the molding from the cavity and takes the form:

$$-\frac{dp}{dz} = \frac{1}{r}\frac{\partial}{\partial r}(r\tau_0)$$

In contrast to the motion equation (9) conduction heat transfer along the OZ axis is substantially due to solidification of the slurry, and heat of phase transition is determined by the apparent heat capacity method (3). In the steady-state solidification process of slurry energy equation can be written as [17–19]:

$$\rho u c_p \frac{\partial t}{\partial z} + \rho v c_p \frac{\partial t}{\partial r} = \frac{\partial}{\partial z}\left(\lambda \frac{\partial t}{\partial z}\right) + \frac{1}{r}\frac{\partial}{\partial r}\left(r\lambda \frac{\partial t}{\partial r}\right) \tag{11}$$

The following notes are used in the Eqs. (9)–(11): z, r – axial and radial coordinates; u, v – components of the velocity vector; p, ρ, T, τ_0, c_p, μ, λ – pressure, density, temperature, shear stress, thermal capacity, viscosity and thermal conductivity of the slurry, respectively.

Condition of the mass flow rate conservation determines the pressure gradient for thermoplastic slurry extrude from the annular cavity [23]:

$$2\pi \int_{r_1}^{r_2} \rho u r dr = \pi \left(r_2^2 - r_1^2\right) \rho_0 u_0 \tag{12}$$

where r_1, r_2 – the radius of the mandrel and bushing, respectively.

Distributions of the velocity and the temperature at the inlet of the cylindrical portion are constant along the cross section of the annular cavity; respectively, all the thermo-physical properties of the slurry are constant:

$$u = u_0, \quad v = 0, \quad t = t_0, \tag{13}$$

if $z = 0$.

On the cavity walls in the area of the liquid slurry state for velocity are put conditions of sticking:

$$u_i = \upsilon_1 = 0, \qquad i = 1, 2 \tag{14}$$

if $z > 0, \quad r = r_i$.

In solid–plastic state they are conditions impermeability and slip:

$$\upsilon_1 = 0, \quad \left(\frac{\partial u}{\partial r}\right)_{r_i} = 0 \tag{15}$$

if $z > 0, \quad r = r_i$.

The assumption is that the heat from the hot slurry is transferred to the walls of bushing and mandrel. Then, condition of heat exchange can be applied to the surface of the mandrel [23]:

$$\lambda \frac{\partial t}{\partial r} = \alpha_d \left(t_c - t_d\right), \tag{16}$$

if $z > 0, \quad r = r_1$.

where α_d – coefficient of heat exchange between the slurry and the wall of the mandrel, t_d – the temperature of the wall of mandrel, t_c – the average temperature of slurry in cross-section of the annular cavity.

If we mark temperature of the water in the hot and cold contours as t_1, t_2, we can put the boundary conditions on the wall of bushing as:

$$-\lambda \frac{\partial t}{\partial r} = k \left(t - t_i\right), \qquad i = 1, 2 \tag{17}$$

if $z > 0, \quad r = r_2$.

where k is the coefficient of heat transfer on the wall of bushing.

At the outlet section of the cavity for temperature are put the following condition:

$$\frac{\partial t}{\partial z} = 0 \tag{18}$$

if $z = l$.

Eqs. (9)–(12) and boundary conditions (13)–(18) are presented in the dimensionless form for convenience. Coordinates z, r are divided by r_1, velocity components u and υ by u_0, pressure p by the value of dynamic head $\rho_0 u_0^2$, temperature t by t_0, density, yield point, coefficients of thermal capacity, viscosity, and thermal conductivity by their values at the temperature t_0.

Set of Eqs. (3)–(12) is solved numerically at boundary conditions of Eqs. (13)–(18) [25]. The considered zone is divided into elementary cells with sides Δz_i, Δr_j. Different analogues of the motion equation (9) and energy (11) were obtained by the Crank–Nicolson method of the second order precision, and different analogue of Eq. (10) was obtained by two layer scheme of the second order precision [23]. Pressure gradient is defined by the splitting method [23] from the condition of conservation of mass flow rate (12).

Coefficients of heat exchange α_d and heat transfer k on the walls of the annular cavity dependent on conductive and convective heat flows, molding velocity, temperature and flow rate of water in the cooling circuits. Heat exchange on the mandrel wall α_d and heat transfer on the bushing wall k were determined by comparison of experimental and calculated data. The inverse problem of an energy equation with unknown heat transfer coefficients at the walls has been solved so that the slurry temperature calculations coincide with the experimental temperature distributions of the slurry. Generalization of experimental and calculated data allows us to determine the dependence of the coefficients of heat transfer and heat exchange from the molding velocity.

4 Results of calculations

The calculation is performed under the same regime parameters and conditions as of the experiments. Calculated data by distribution of temperature in an annular cavity according to the conditions of the first series of experiments are demonstrated in Fig. 4 (Table. 1).

At the inlet of the cylindrical part of the annular cavity the temperature of the slurry is constant and equal to t_0=80°C. In the area of hot contour isolines (isotherms) of the temperature show the zones of the constant values of the temperature and parameters of the slurry mass is in liquid state. In this part the temperature of the slurry and the hot water is the same, heat transfer practically does not occur on the bushing wall.

Cooling of the slurry mass starts in the area of cold contour. Difference of the temperature of the slurry mass and cold water leads to an intensive heat transfer in the second cooling contour, and to reduction of the temperature and to change of rheological properties of the slurry.

The slurry temperature field is variable and changes from 80 to 54°C in the beginning of the second contour.

Isotherm with the temperature 54°C expresses upper bound of solidification zone of the slurry mass, and isotherm 40°C expresses the lower bound of the solidification zone. In the area of solidification between isotherms 54 and 40°C the slurry passes from the liquid (viscous–plastic) state to solid–plastic state. The experimental data of isotherms "solidus" AB (54°C) and "solidus" CD (40°C) are shown in Fig. 2 and 3. It may be noticed an agreement between the calculated data and experiments of positions of isotherms AB and CD.

At the value of the molding velocity of u_0=20 mm/min position of the transition zone of the slurry from the liquid (viscous-plastic) state to solid-plastic state is located closer to the beginning of the cold contour of cooling. With increasing molding velocity from 40 to 100 mm/min the position of the transition zone begins to pull down towards movement of the molding and takes extensive areas. It explains that with increasing of molding velocity convective component of heat flow of the slurry mass increases. The position of the transition zone increases and it covers all length of the mold cavity (Fig. 4).

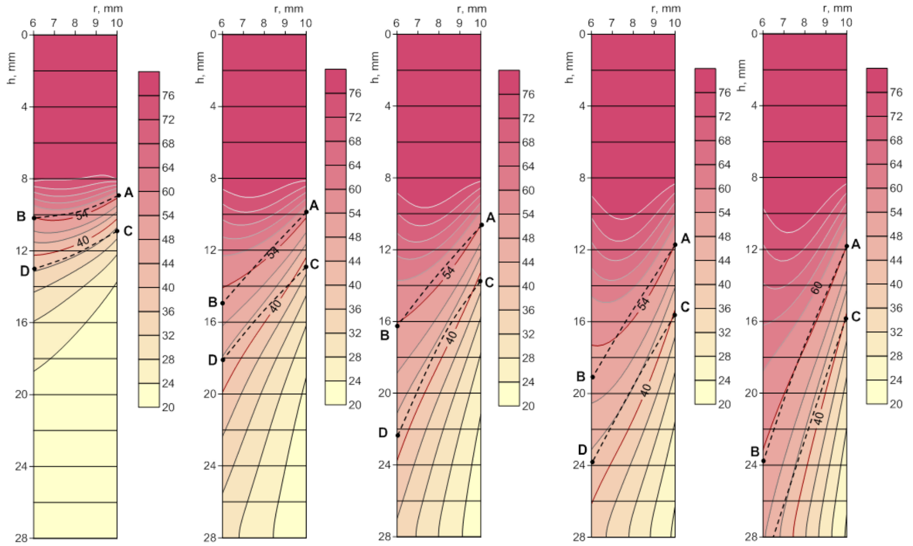

Fig. 4. Comparison of the calculated and experimental data of distribution of the temperature depending on the molding velocity (Table. 1).

The effect of flow rate and temperature of cold water on the position of the transition zone of the molding by conditions of the experimental researches were determined in the second series of calculations (Table. 2). Calculation data of the temperature distribution and the position of the transition zone of molding, limiting by isotherms ”solidus” AB and ”solidus” CD are shown in Fig. 5. In the first three cases, the temperature of cold water is 15°C, and its flow rate reduces from 1000 to 250 l/hour, respectively. Reducing the temperature of cold water increases the intensity of heat extraction and reducing of its flow rate and vise versa, it slows down the process of heat extraction. Increasing the temperature of cold water till 20°C, as well as reducing of its flow rate leads to a reduction of heat extraction.

The calculated temperature data are in agreement with the experiment results (Fig. 5).

The experimental and calculated data show that beryllia thermoplastic slurry solidification process can be controlled by adjusting the flow rate and temperature of the cold water.

5 Conclusion

During the series of experiments of the research on the effect of casting regimes on the temperature field in the solidification zone of the molding were identified the followings:

- the position of solidification zone of the slurry mass when molding velocity changes from 20 to 100 mm/min;

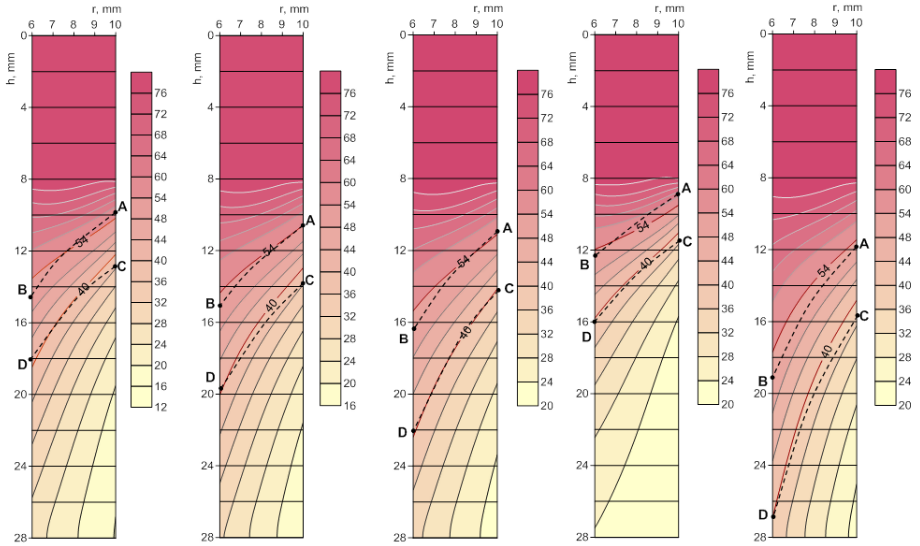

Fig. 5. Comparison of the calculated and experimental data of distribution of the temperature depending on the flow rate and the temperature of cold water (Table. 2).

- the position of solidification zone of the slurry mass in the form-building cavity depending on the water flow rate and the temperature in the cold contour of cooling.

Temperature distribution, estimated during the experiments, in the form-building cavity of bushing depending on the molding velocity and heat extraction conditions on the walls of form-building of annular cavity lets us determine the transition from liquid (viscous-plastic) state to solid-plastic one.

The experiment results were analyzed and generalized using mathematical model of the thermoplastic slurry molding process. The latent heat of the phase change has been accounted by the apparent heat capacity.

The mathematical model includes the equations of the law of conservation of mass, momentum and energy of non-Newtonian fluid with the Shvedov-Bingham's rheological model. Rheological and thermo-physical properties of the slurry were found on the basis of experimental data and express dependence on the temperature. The coefficients of heat exchange and heat transfer on the walls of the annular cavity were determined by comparison of experimental and calculation data. The temperature field of the slurry in liquid (viscous-plastic) and solid-plastic states were obtained in the calculations. The positions of isotherm "solidus" (54 °C) and "solidus" (40 °C), expressing the upper and lower boundaries of the solidification zone position were determined.

The results of calculation are in agreement with the experimental data, and they show physical validity of the proposed mathematical model of the molding process of the beryllia thermoplastic slurry.

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